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54) Process for producing modified oxide layers

57) The invention comprises a process for producing modified oxide layers on substrates coated with aluminium metals, said substrates being able to be both metallic and non-metallic in nature. Said oxide layers can be oxidised both partly and completely, e.g. by anodic oxidation with spark discharge, and exhibit in particular with complete oxidation an outstanding adhesion, on both metallic and non-metallic substrates. The oxide layer itself is roughened on the surface and is a good adherend surface for subsequent coatings. In the case of oxidised metal substrate-oxide layer combinations the corrosion resistance is also influenced positively at temperatures > 923 K. A good electrical insulation is achieved by means of the modified oxide layers on metallic substrates, with simultaneous improved thermal conduction of the oxide layer. A noticeable increase in insulating power is also demonstrated on organic substrates, e.g. epoxy resins, and the high-voltage characteristics are also improved. The composites can be used in machine and vehicle construction, in the chemical and electronics industry [1].

[1] There are some two to three words missing in the original here. – Translator

Process for producing modified oxide layers

Scope of the invention

The invention relates to a process for producing modified oxide layers on substrates coated with aluminium metal which can be of both metallic and organic structure. By means of this process the oxide layers can be oxidised partly or completely, which results in their selective use in electrical engineering/electronics, in machine and vehicle construction and in the chemical industry. Since said modified oxide layers adhere well to the substrate and because of their structure exhibit an improved adherend surface, they can be coated with good adhesive effect in particular with paints, insulating materials etc. and be easily formed. The layers produced according to the invention are because of their good corrosion-proofing effect, which is also retained at high temperatures, usable in vehicle construction, in particular for exhaust systems. In the chemical industry the oxide layers produced according to the invention can be used as support materials mainly for catalysts or as reactor linings. Because of their high electrical resistance properties and good thermal dissipation, they can be used in particular in the electrical engineering and electronics sector.

Characteristics of the known technical solutions

Processes for coating metals and non-metallic materials with Al_2O_3 from the gas phase or directly by plasma spraying are known. The oxide layers so produced are either too thin or brittle and mechanically unstable or they show with greater layer thicknesses an insufficient adhesion and unsatisfactory forming characteristics. In many cases said layers exhibit an excessively smooth surface morphology, so that they are unsuitable as adhesion promoters e.g. for paints, dyes and other coating systems, and as insulating material supports. Processes for producing thicker layers, which are produced e.g. by plasma spraying of Al_2O_3 onto metallic materials, show that said oxide layers are insufficiently compact and cannot therefore be used for a permanent corrosion proofing or for electrical insulation purposes. Furthermore, the plasma spraying of Al_2O_3 cannot be applied to organic substrates.

Aim of the invention

The aim of the invention is to produce, by a novel process, modified oxide layers on metallic and non-metallic substrates which exhibit an improved adhesive strength on the substrate, and

also form a good adherend surface for paint and insulating substance systems and show improved forming, corrosion-proofing, electrical resistance and heat conduction properties.

Description of the gist of the invention

The invention is based on the object of developing a process for the production of modified oxide layers on substrates coated with aluminium metals. According to the invention the object is achieved by the fact that a composite, e.g. steel coated with aluminium, which is produced by aluminizing or the electrochemical deposition of aluminium on steel in non-aqueous solutions, is subjected to an anodic oxidation with spark discharge in aqueous electrolytes, according to DDWPC25D/211430. The aluminium layer is here partly or completely oxidised, and because of the high temperatures at the surface of the aluminium the oxide layer is enriched with α-Al_2O_3. Due to the high temperatures in the discharge channel a plasma state is generated, which melts the forming Al_2O_3 on the substrate surface and produces an improved adhesion of the resulting coating. The greatest adhesion is achieved if the aluminium layer on the substrate is completely oxidised and forms a strong chemical bond with the substrate surface. Said bond can be achieved in the case of metallic substrates via intermediate layers, e.g. with spinel structure, which arise from a reactive substrate-oxide layer interaction. In the case of organic substrates a roughening of the substrate surface occurs, and at the same time the adhesion of the Al_2O_3 layer to the substrate is increased significantly according to the "push-button mechanism".

It was found by pull-off tests that the adhesive strength comes to approx. 26 MPa in the case of partly oxidised substrate-oxide layer combinations, and > 30 MPa in the case of completely oxidised substrate-oxide layer combinations.

In the case of completely oxidised substrate-oxide layer combinations the corrosion resistance is influenced positively at temperatures > 923 K.

As a result of the modified oxide layers on metallic substrates produced according to the process an improved electrical insulation is achieved with simultaneous improved thermal conduction.

On organic substrates, e.g. epoxy resins etc., an improvement of the insulation characteristics is demonstrated, which particularly in the case of a substrate-oxide-substrate combination shows itself in improved high-voltage characteristics.

Embodiment

The invention will be explained below from two embodiments.

Embodiment 1

An anodic oxidation with spark discharge is carried out on an iron sample which is coated to approx. 10 µm with aluminium. In so doing the aluminium is completely oxidised and remains as a firmly adhering, molten and compact oxide layer on the substrate surface. Pull-off tests to determine the adhesive strength show values of > 30 MPa on this layer. If, by variation of the oxidation parameters, the 10 µm thick aluminium layer on the present iron sample is oxidised to a depth of only 6 µm by means of anodic oxidation with spark discharge, an adhesive strength of the oxide layer on the aluminium base of ~ 25 MPa is obtained.

Embodiment 2

An anodic oxidation with spark discharge is carried out on an epoxy resin sample, which is coated with a 5 µm thick aluminium layer, up to complete oxidation of the aluminium. The oxide layer is coated with a further 6 – 8 µm epoxy resin coat by conventional post-treatment methods. Measurements of the d.c. voltage disruptive field strength show a ~ 20% higher loading capacity compared with pure epoxy resin samples.

Claim

1. Process for producing modified oxide layers using anodic spark discharge, characterised in that a substrate-oxide layer composite is produced which shows a high adhesive strength and is corrosion-resistant, electrically insulating and highly thermally conductive.

2. Process according to claim 1, characterised in that the substrate is both a metallic and a non-metallic material.

3. Process according to claims 1 and 2, characterised in that the substrate is an organic material.

4. Process according to claims 1, 2 and 3, characterised in that the metal component to be oxidised on the substrate material is oxidised partly or completely.